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Ultra-high performance concrete made from the insertion of glass residues as supplementary cement material

Concretos de ultra alto desempenho confeccionados a partir da inserção de resíduos de vidros como material cimentício suplementar

Hormigones de ultra alto rendimiento fabricados con a partir de la inserción de residuos de vidrios como material de cemento suplementario

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Abstract
Portland cement concrete manufacturing techniques in the world have an immense variability, since each location concentrates natural resources with unique characteristics, sometimes similar, but with always different process conditions, such as: labor, equipment, environmental conditions, processing, storage, among others. The aim of this work is to develop cementitious composites partially replacing mineral admixtures by powdered glass residues, evaluating the physical-mechanical behavior through axial compression strength, as well as the production of hydrated phases through x-ray diffraction, both at 28 days. Cement, silica fume, metakaolin, powder glass residue, polycarboxylic ether-based superplasticizer, and low temperature water were used to prepare different compositions of ultra-high performance concrete. The preparation took place with the aid of a mechanical bench mixer, and the densification was carried out with the aid of an immersion vibrator in cylindrical molds 50 x 100 mm. After unshaped, the specimens received heat treatment, with isotherm at 60°C for 36 hours, followed by wet curing in a tank saturated with calcium hydroxide, until the tests of compression strength and x-ray diffraction were carried out. The results show that the composition added with the residue, replacing metakaolin, behaved in an adequate manner, with compressive strength equivalent to the reference composition (silica fume + metakaolin), as well as the production of the same hydrated compounds. However, the application of only the glass residue or metakaolin reduced the intensity of the pozzolanic reactions, justified by the presence of a high content of calcium hydroxide in the compositions, which allows us to conclude that the powder glass residue presents viability as a filler, when replacing metakaolin in the presence of silica fume, as it does not compromise the performance of the mixtures made.
Keywords: Glass Residue; UHPC Systems; Sustainability.

Resumo
As técnicas de fabricação de concreto de cimento Portland no mundo possuem uma variabilidade imensa, uma vez que cada localidade concentra recursos naturais com características únicas, as vezes similares, mas com condições de processo sempre diferentes, como: mão de obra, equipamentos, condições ambientais, processamento, armazenamento, entre outros. Objetiva-se com esse trabalho desenvolver compósitos cimentícios substituindo parcialmente as adições minerais por resíduos de vidro em pó, avaliando o comportamento físico-mecânico por meio de resistência à compressão axial, bem como a produção de fases hidratadas através de difração de raios-x, ambos aos 28 dias. Utilizou-se cimento, microssílica, metacaulim, resíduo de vidros, superplastificante de base éter policarboxílico, e água em baixa temperatura para confeccionar diferentes composições de concreto de ultra alto desempenho. A preparação ocorreu com auxílio de misturador mecânico de bancada, e o adensamento foi realizado com auxílio de vibrador de imersão em moldes cilíndricos 50 x 100 mm. Após desmolde, os corpos de prova receberam tratamento térmico, com isoterma à 60°C por 36 horas, seguido de cura úmida em tanque saturado de hidróxido de cálcio, até a realização dos ensaios de resistência à compressão e difração de raios-x. Os resultados

apontam que, a composição aditivada com o resíduo, substituindo o metacaulim, comportou-se de forma adequada, com resistência à compressão equivalente à composição de referência (microssílica + metacaulim), bem como a produção dos mesmos compostos hidratados. No entanto, a aplicação de apenas o resíduo de vidro ou metacaulim, reduziu a intensidade das reações pozolânicas, justificado pela presença de elevado teor de hidróxido de cálcio nas composições, o que permite concluir que o resíduo do vidro em pó apresenta viabilidade como fíler, ao substituir o metacaulim na presença de microssílica, por não comprometer o desempenho das misturas confeccionadas.
Palavras-chave: Resíduo de Vidro; Sistemas CUAD; Sustentabilidade.

Resumen
Las técnicas de fabricación de concreto de cemento Portland en el mundo tienen una inmensa variabilidad, ya que cada ubicación concentra recursos naturales con características únicas, a veces similares, pero con condiciones de proceso siempre diferentes, tales como: mano de obra, equipos, condiciones ambientales, procesamiento, almacenamiento, entre otros. El objetivo de este trabajo es desarrollar composites cementosos mediante la sustitución parcial de adiciones minerales por residuo de vidrio en polvo, evaluando el comportamiento físico-mecánico mediante resistencia a la compresión axial, así como la producción de fases hidratadas mediante difracción de rayos X, ambos a 28 días. Se utilizaron cemento, sílice activa, metacaolín, residuos de vidrio, superplastificante a base de éter policarboxílico y agua a baja temperatura para hacer diferentes composiciones de hormigón de ultra alto rendimiento. La preparación se realizó con la ayuda de un mezclador mecánico de mesa y la densificación se realizó con la ayuda de un vibrador de inmersión en moldes cilíndricos de 50 x 100 mm. Después del desmoldeo, las probetas recibieron tratamiento térmico, con isoterma a 60 °C durante 36 horas, seguido de curado húmedo en un tanque de hidróxido de calcio saturado, hasta que se realizaron las pruebas de resistencia a la compresión y difracción de rayos X. Los resultados muestran que la composición añadida con el residuo, en sustitución del metacaolín, se comportó correctamente, con una resistencia a la compresión equivalente a la composición de referencia (sílice activa + metacaolín), así como la producción de los mismos compuestos hidratados. Sin embargo, la aplicación de solo el residuo de vidrio o metacaolín, redujo la intensidad de las reacciones puzolánicas, debido a la presencia de un alto contenido de hidróxido de calcio en las composiciones, lo que nos permite concluir que el residuo de polvo de vidrio es viable como relleno, al reemplazar el metacaolín en presencia de sílice activa, ya que no compromete el rendimiento de las mezclas realizadas.
Palabras clave: Residuos de Vidrio; Sistemas HUAR; Sustentabilidad.

1. Introduction

The Ultra-High Performance Concrete (UHPC) technology emerged in the last 25 years through a partnership between Canadian and French researchers, based on theoretical models of particle packing, exclusion of coarse aggregates, use of heat treatment at high temperatures, tension compression to the mixture in the fresh state, very low w/c factor, use of additives and mineral admixtures, and application of fibers or microfibers (Soliman & Tagnit-Hamou, 2017a; Kang, Hong & Moon, 2019; Cunha Oliveira, Meira & Lucena, 2021a).

Richard & Cheyrezy (1995), reached strengths of the order of 810 MPa in analysis of compositions for Reactive Powder Concrete (RPC), one of the UHPC variants, using steel aggregate and heat treatment at 250 °C.

Studies dating back to 1930 already suggested the use of some of these devices to increase the strength of the paste, and in the 1960s laboratory evaluations showed the reach of strengths close to 650 MPa when compressing the mixture while still in the fresh state with the simultaneous use of treatment thermal (Richard & Cheyrezy, 1995).

The UHPC class is widely developed in the world, and because it is a recent technology, engineers always use different methodologies for each application due to the lack of standardization standards, thus creating different types of concrete, varying according to the available raw material, level of specialization of labor, equipment for manufacturing and execution, and demand for use.

The technology has been disseminated mainly in European and Asian countries, and in Brazil it is still impracticable to apply this type of concrete on a large scale due to the lack of qualified labor and adequate equipment for confection, as well as the high cost of raw material. However, what will guide the feasibility of use in Brazilian works in the coming decades will possibly be the adoption of cheaper execution techniques associated with the use of locally sourced materials, both in line with sustainable work streams.

UHPC has become an alternative among concretes available for structural use (conventional and high performance), due to the excellent mechanical properties obtained from the high density of the hydrated matrix of the material.

The excellent durability acquired after the curing procedure at high temperatures, allows the useful life of the composite to reach marks that easily exceed 100 years. Matte (1999) states that it is possible to reach 300 years by estimating a depth of degradation of 1.4 cm. In this way, it is even possible that, in certain situations, the use of negative reinforcement is suppressed due to the insertion of metallic fibers (by increasing the tensile strength, ductility and toughness of the composite).

Since the idealization of RPC by Richard & Cheyrezy (1995), which optimized the basic principles of UHPC composition from a theoretical model of particle packing and the use of heat treatment under pressure. Lafarge Holcim has patented the material as Ductal®, which has been on the European market since 2001, being exported all over the world with the possibility of different applications, whether in architecture, infrastructure and superstructures, paving sector, furniture for outdoor areas, construction elements such as brise-soleil panels, among others.

Linked to the elaborate dosing process, the resulting costs per unit of m^3 of UHPC make it unfeasible for small-scale applications, but the cost-benefit is attractive when the works are large, such as bridges, industrial environments, or even structures in maritime areas , as among the factors that most encourage the use of this technology, the significant reduction in maintenance costs is the most highlighted, since its useful life can be, on average, three times longer than that of conventional concrete (150 years).

In a higher class of strength and durability, RPC was created by Richard & Cheyrezy (1995), classified among the ultra-high-performance concretes based on Portland cement that has the best physical and mechanical properties patented to date, used in same applications as conventional concrete or as auxiliary technology. The minimum threshold for compressive strength comprises values of the order of 120 MPa (Dixit, Du & Pang, 2020), however, in most studies, 150 MPa is adopted at 28 days of age (Kang, Hong & Moon, 2019; Liu, Wei, Zou, Zhou & Jian, 2020; Sohail, Kahraman, Nuaimi, Gencturk & Alnahhal, 2021).

However, for Cunha Oliveira, Meira & Lucena (2021a), in scale, conventional concrete can achieve strengths of up to 60 MPa, high performance concrete between 60 MPa and 120 MPa, and UHPC encompasses a range of compressive strength ranging from 200 MPa to 800 MPa, subdivided into classes of UHPC 200 and UHPC 800, which depending on the resistance range, new classifications are used, such as the UHPC C500.

According to Dong (2018), while conventional concrete has an average cost of US$ 100/m^3, ultra-high performance can reach US$ 830/m^3. One of the ways to make the technology economically viable is to use cheaper inputs in its composition. Yang *et al.* (2020), managed to reduce the final cost of a UHPC by 42%, without harming the mechanical strength, applying stone dust as recycled aggregate.

In this way, it is permissible to reduce the real costs with this technology, and Soliman & Tagni-Hamou (2016) also point to savings through the replacement of quartz aggregate (non-renewable raw material) by waste glass, due to the fact of this type of recycled aggregate is three times cheaper.

Bearing in mind that UHPC systems use high percentages of mineral admixtures to increase their performance in terms of axial compression and durability (Cunha Oliveira, 2020), due to the pore refinement on a microstructural scale resulting in high compactness (Cunha Oliveira, Meira & Lucena, 2021a), the glass residue incorporated into the composition as supplementary cementitious materials (SCM) (filler), due to reduced particle size, recommends its use for particle sizes below the fine aggregates, because the specific mass is similar and has less absorption (Soliman & Tagnit-Hamou, 2017a).

Verifying the presence of pozzolanic behavior in industrial by-products makes it possible to classify whether SCM are inert (filler) or reactive (pozzolan) (Cunha Oliveira, Meira & Lucena, 2021b). The pozzolanic potential of a material is

configured by detonating cementing properties when it reacts in the presence of calcium hydroxide (C-H), which originates from the hydrated phases of Portland cement, producing low-density calcium silicate hydrated (C-S-H) (Cunha Oliveira, Chagas, Meira, Carneiro & Melo Neto, 2020; Cunha Oliveira, Meira & Lucena, 2021a), similar to that produced by C_3S and C_2S (tricalcium and dicalcium silicates), and which increases the durability of the cement matrix in the hardened state.

According to Jiang, Ling, Mo & Shi (2019), the reuse of glass waste in the construction industry not only reduces the burden on landfills, but also significantly contributes to the preservation of resources and reduction of CO_2 emissions. Applying glass residue in cementitious matrix composites as an alternative material has been a long-established practice (Zhang, He & Poon, 2020), and which makes it possible, for example, to reduce the quantities of Portland cement or aggregate/powder from quartz in compositions.

The objective of this work is to investigate the mechanical behavior of ultra-high performance concrete specimens, at 28 days of age, with different amounts of powdered glass residue as mineral admixtures, and in parallel, identify the hydrated phases formed in the composites by means of x-ray diffraction, correlating these with resistance values to evaluate the behavior of different compositions of UHPC systems through the principle of pozzolanicity of the SCM.

2. Methodology

2.1 Processing of Glass Residue

The soda-lime-silicate glass residue, originating from the disposal of bottles, packaging, and household utilities, of various colors, was collected and sanitized to remove most impurities, to be later processed, which took place in the Comminution Laboratory of the IFPB – Campus Campina Grande. The first step consisted of crushing the vitreous waste using a jaw crusher (Figure 1a). Therefore, the excess impurities (metals, pieces of paper) were removed and the crushed residue was milled in a bench ball mill, at 47 rpm, adopting an interval of 3 hours for each grinding cycle (Figure 1b).

Figure 1 – Jaw Crusher (a), and Ball Mill (b).

Source: Authors.

Finally, the ground residue was sieved in a 75 μm sieve (ABNT nº 200) with the aid of a mechanical stirrer in cycles of 30 minutes at a frequency of 8 Hz (Figure 2). All material passing through the mesh was stored in plastic bags to be used in

the manufacture of UHPC specimens, and the retained material was taken to grinding in the ball mill.

Figure 2 – Screening in ABNT n° 200 mesh (a), and final visual appearance of the residue (b).

Source: Authors.

2.2 Production of Ultra-High Performance Concretes

Table 1 shows the six formulations of the composites that will be studied, in mass proportions: SF+MK (reference), SF+PGR, MK+PGR, PGR100, MK100 and SF100, where SF corresponds to the presence of silica fume in the composition, MK designates metakaolin, and PGR the powder glass residue.

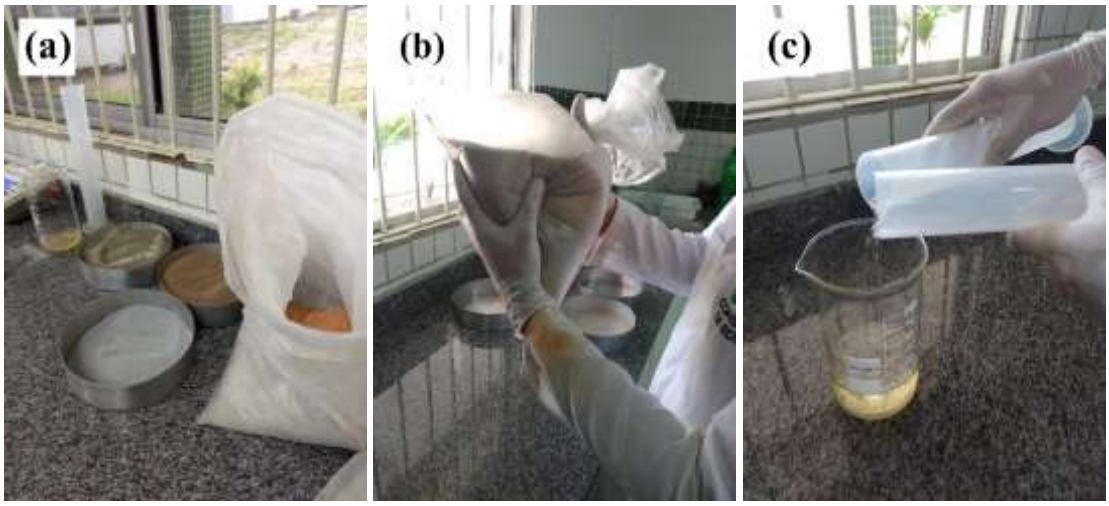
Table 1 – Mass proportions for making UHPC systems.

Materials	SF+MK	SF+PGR	MK+PGR	PGR100	MK100	SF100
Cement	1.000	1.000	1.000	1.000	1.000	1.000
Silica Fume	0.110	0.110	–	–	–	0.170
Metakaolin	0.060	–	0.060	–	0.170	–
Powder Glass Residue	–	0.060	0.110	0.170	–	–
Sand	1.050	1.050	1.050	1.050	1.050	1.050
PCE	0.075	0.075	0.075	0.075	0.075	0.075
w/b	0.170	0.170	0.170	0.170	0.170	0.170

Source: Authors.

Initially, after weighing the raw materials, all powdered materials (cement, sand, silica fume, metakaolin and powder glass residue) were pre-mixed with the aid of plastic bags, for 3 minutes, before adding water and of the superplasticizer additive (PCE), to minimize errors related to compositional imbalance, as well as to avoid agglomeration of particles (Soliman & Tagni-Hamou, 2017b), while the PCE was diluted in the kneading water, in order to obtain a single solution (Figure 3). The procedure was performed in Construction Materials Laboratory of the IFPB - Campus Campina Grande.

Figure 3 – Weighing of raw materials (a), and homogenization of powders (b) and liquids (c).



Source: Authors.

All compositions of the UHPC systems were tested in a benchtop mechanical mixer with a capacity of 4.5 L. The homogenization procedure, based on the UHPC manufacturing methodologies adopted by Hiremath & Yaragal (2017), and Wang *et al.* (2018), occurred as follows: approximately 50% of the liquid (water + PCE) and 40% of the powdered materials were added to the mixer and homogenized for 5 minutes at high speed, and subsequently, the rest of the inputs were gradually added to the vat, in 4 steps, with each lasting 3 minutes, all at low speed, to avoid loss of raw material and ensure agglutination of the particles during homogenization. In the last step, the mixture remained for 5 minutes in the mixer at high speed after the gradual addition of the remaining powders and liquid (Figure 4).

Figure 4 – Pause between steps for adding inputs (a), and final visual appearance (b).

Source: Authors.

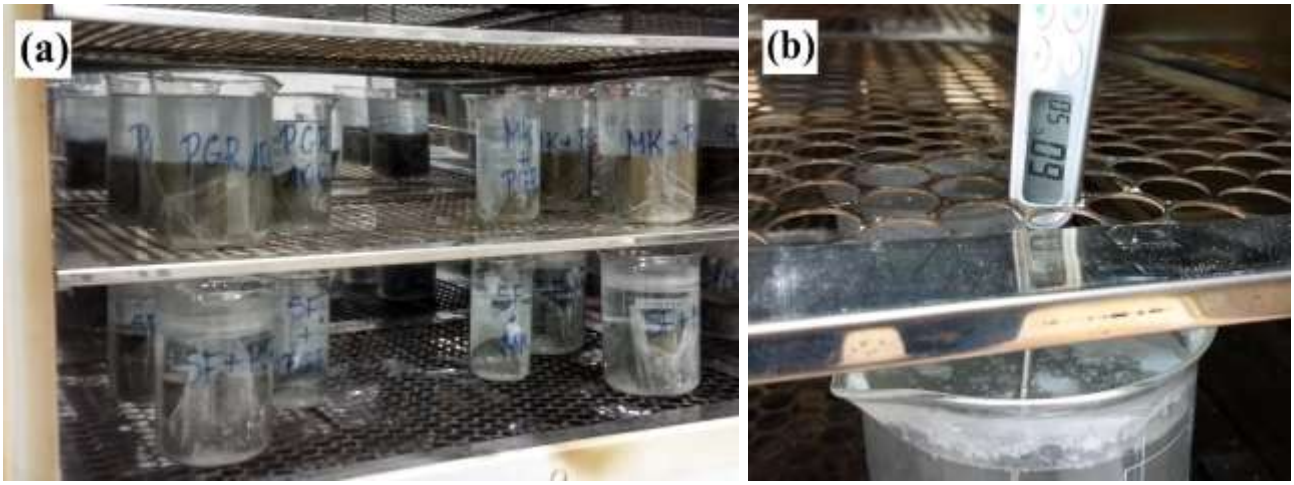
For each composition, six cylindrical specimens measuring 50 x 100 mm were molded to perform the mechanical test, as well as to evaluate the hydrated phases by means of x-ray diffraction. The densification of the cylindrical specimens followed the procedures prescribed by ABNT NBR 5738:2015, and was carried out with a portable immersion vibrator VIBROMAK Model VBP-2512, with a frequency of 175 to 210 Hz and a needle diameter of 25 mm. The application of the vibration was divided into 2 layers of 50 mm, with a needle dwell time of 25 seconds per layer as recommended by Bauer

(2019). Then, the specimens were covered with plastic film until demolding was performed (Vanderlei, 2004), to ensure that the water, in the first 24 hours at room temperature, was not lost.

The curing procedure was performed with partial support in the study by Vanderlei (2004), following the steps: with the aid of beakers, the specimens were immersed in drinking water at room temperature (25 °C), and remained for 4 hours until heat treatment is started. In total, adding to the 24 hours elapsed after confection, the waiting period, or thermal pre-cure time, of 28 hours, has been completed.

Consequently, the specimens submerged in the beakers were conditioned in a SOLAB Model SL-100 electric oven with a power of 3,000 W and forced air circulation, at a heating rate of 0.15 °C.min^{-1}, to water reach a temperature of 60 °C, remaining in isotherm for 36 hours. Warming occurred gradually by induction, and lasted 4 hours until it remained stable (Figure 5).

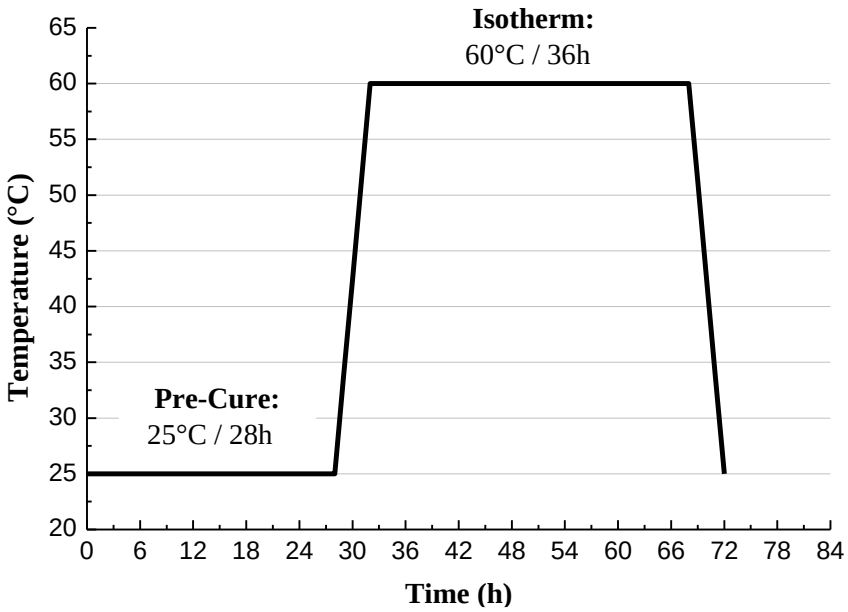
Figure 5 – Beakers in an electric oven (a), and temperature measurement during isotherm (b).



Source: Authors.

After completing the isotherm time, which ensured the total submersion of the specimens throughout the period, the beakers were removed from the electric oven and placed on a bench at room temperature, to cool slowly without any possible thermal shocks to the matrices of the UHPC systems, which took about 4 hours. The total treatment time was 44 hours (heating + isotherm + cooling), comprising the second stage of heat treatment (Figure 6).

Figure 6 – Curing regime used for making UHPC systems.



Source: Authors.

After the heat treatment was completed, the specimens were submerged in a saturated solution of water + calcium hydroxide ($Ca(OH)_2$), which corresponds to wet curing, at room temperature, remaining under this condition until completing 28 days to perform the microstructural scale testing as well as mechanical testing (Figure 7).

Figure 7 – Cylindrical specimens immersed in water + Calcium hydroxide ($Ca(OH)_2$).

Source: Authors.

2.3 Axial Compression Strength Evaluation

The efficiency in the production of hydrated compounds was measured through the mechanical behavior of the UHPC matrices, breaking the specimens at 28 days of age, in the Construction Materials Laboratory of the IFPB – Campus Campina Grande. MATEST Model 100T hydraulic press was used, with test performed according to ABNT NBR 5739:2018. The standard recommends that the loading be applied perpendicularly to the cylindrical specimen, with a controlled breaking load

speed of 0.45 MPa/s (± 0.15), constant throughout the test and without shocks.

2.4 Identification of Hydrated Phases

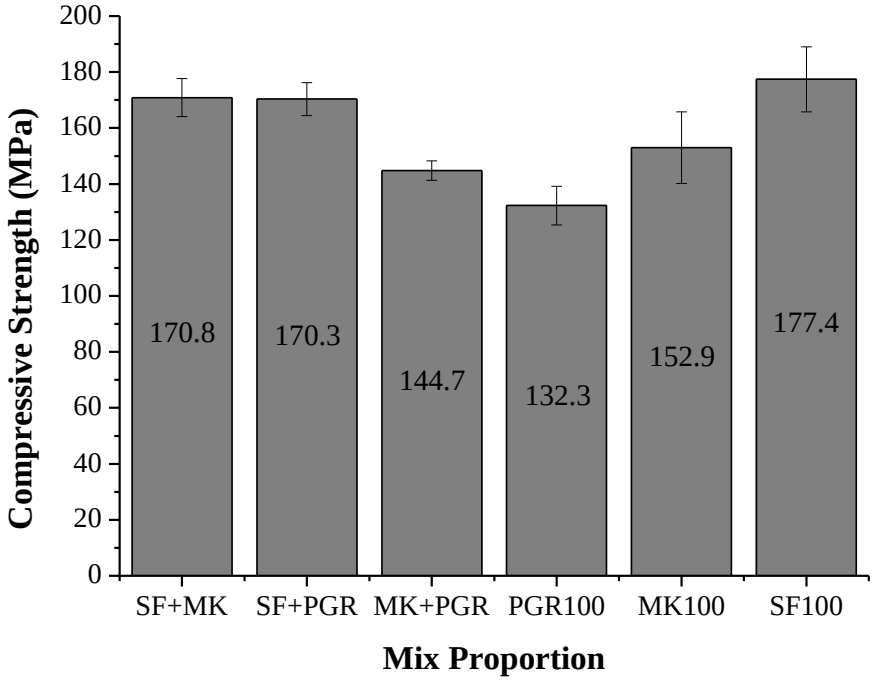
The X-Ray Diffraction (XRD) test was carried out at the Materials Characterization Laboratory (CCT/UAEMa), at UFCG – Campus Campina Grande, using the powder method. The SHIMADZU Model XRD-6000 equipment (X-Ray Diffractometer) was used, which has a fixed x-ray tube in high vacuum with tungsten filament, with the sample rotation in θ and the arm in 2θ. The radiation used was Kα (monochromatic) for the target metal Copper (Cu), with λ = 1.5406 Å, under a voltage of 40 kV and a current of 30 mA, with slits opening at 1.0°, 1.0° and 0.3 mm. The scanning speed was set at 2°.min^{-1}, at a sampling step of 0.02° and a stop time of 0.60 s, in the range of 5° to 60°.

3. Results and Discussion

3.1 Axial Compression Strength Evaluation

Figure 8 below shows the mechanical behavior of the developed compositions, where different amounts of mineral admixtures were added in the UHPC systems, broken at 28 days of age.

Figure 8 – Mechanical behavior of UHPC compositions at 28 days of age.



Source: Authors.

It is observed that, initially, four of the six compositions surpass the minimum value of 150 MPa at 28 days of age, expressed in the literature, to qualify as UHPC systems (Kusumawardaningsih, Fehling, Ismail & Aboubakr, 2015). However, it can be considered that the minimum of 120 MPa is already enough to classify the mixtures as UHPC (Dixit, Du & Pang, 2020), which is correct if the way of making the mixtures is emphasized, in addition to the value be above the usual for high performance concrete. In other words, all compositions are in accordance with UHPC technology when considering axial compressive strength values at 28 days.

Regarding the mechanical behavior between the compositions, the SF+MK (reference) composition presented 170.8 ±

4.8 MPa, using silica fume and metakaolin, while the SF+PGR reached 170.3 ± 3.9 MPa, value equivalent to the reference, showing that the adoption of waste glass does not compromise the mechanical behavior when metakaolin is replaced by the same.

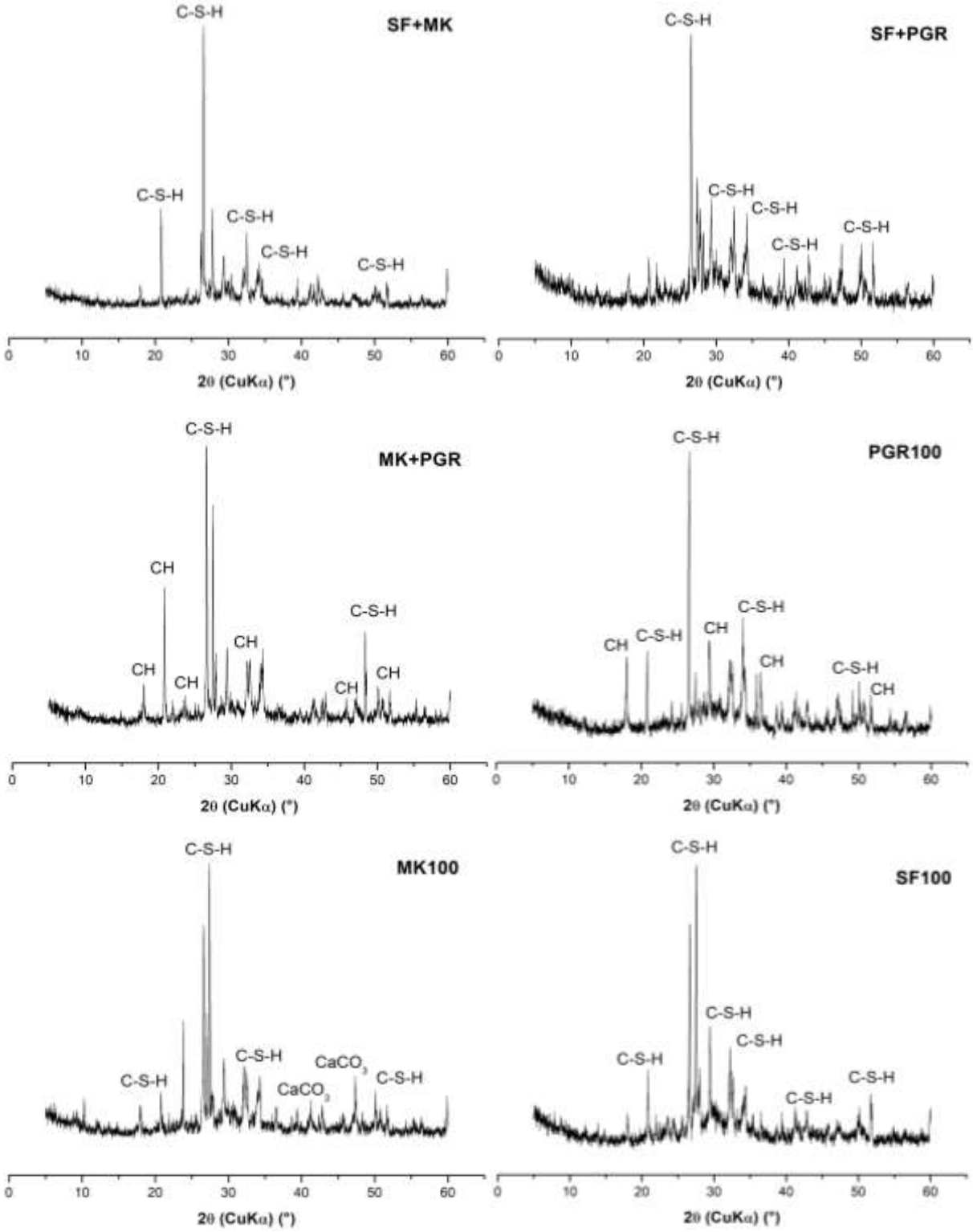
In the opposite situation, the MK+PGR composition presented a value of only 144.7 ± 3.4 MPa, 15% lower than the reference, and this drop in resistance is attributed to the non-consumption of C-H during the pozzolanic reactions during hydration of the composite, and in addition, the size distribution of metakaolin is different, as well as the particle shape, the interaction between hydrated phases is also different when comparing the behavior with silica fume, leaving more voids in the gel pores of the mixture. Tafraoui, Escadeillas & Vidal (2016) in their experiments obtained similar results for different UHPC compositions, which used the two mineral admixtures, with the metakaolin additive composition presenting inferred value compared to the silica fume composition, being directly related to the effective consumption of C-H from the paste for the production of C-S-H.

Among the three compositions that used only one mineral admixtures as an input (PGR100, MK100, SF100), it is noted that SF100 presented 177.4 ± 8.6 MPa, being the highest value among the three that were made, which again confirms the pozzolanic efficiency of silica fume compared to metakaolin, which obtained only 152.9 ± 9.7 MPa. The composition that only incorporated powder glass residue as SCM presented 132.3 ± 5.8 MPa, being the lowest value among all the compositions, which demonstrates the non-reactivity of this supplementary cementitious material, as the low strength confirms the low production of C-S-H arising from pozzolanic reaction, with the mineral admixture behaving like a filler, only refining the capillary pore structure of the material.

3.2 Identification of Hydrated Phases

Figure 9 below shows the compositions of the mineralogical compositions UHPC, obtained by XRD test at 28 days age.

Figure 9 – Mineralogical composition of UHPC compositions at 28 days of age.



Source: Authors.

As expected, it was observed in the diffractograms the presence of portlandite (C-H) in the compositions that showed lower compressive strength at 28 days, confirming that the compound released during the hydration of the composites was not consumed by the powder glass residue (PGR100), in higher intensity in the diffractogram, followed by lower intensity for the MK+PGR, which was not effective in producing low-density C-S-H in the presence of C-H, but which compared to the

PGR100, consumed more C-H because the peak in the diffractogram was lower, as well as its incidence, which was in a smaller proportion.

It was noted that for MK100 calcium carbonate ($CaCO_3$) was produced during the hydration of the composite, which is attributed to the dissociation of carbon dioxide, which after reacting with C-H releases carbonates and water (Medina, 2011; Santos, Albuquerque & Ribeiro, 2020). For an initial approach, it was noted that in general, all compositions that presented C-S-H in their composition may have opportunely presented mineralogical phases corresponding to the concentration of hydrated calcium silicates, which in the presence of other mineralogical constituents, may have formed other hydrated phases. In parallel, the lack of evidence of C-H in the other pastes does not mean that it is not present, even if in an undetectable proportion by XRD.

4. Conclusion

As for the mechanical behavior of the compositions, it was observed that they all fit within the expected range for UHPC mixtures, with dosage sizing, mixing procedure and thermal cure proving adequate for the production of composites.

The compressive strength results of the composites were conclusive attesting that the powder glass residue behaved properly when inserted in the compositions, both together with metakaolin and silica fume. However, despite the reduction in strength when the residue is added alone to the Portland cement matrix, it was noted that the value obtained for the test is in accordance with the class of UHPC systems, which demonstrates technical, economic and mainly ecological feasibility.

In view of the results of the X-ray diffractometers, it was observed that the formation of C-S-H in the compositions was effective where the microsilica was together with the residue, or with the metakaolin, or when it was added in an isolated form. When metakaolin was inserted together with the residue, the C-H was not totally consumed, in the same way when the powder glass residue was added alone in the UHPC matrix.

Correlating the parameters, it is observed that the reduction in the compressive strength values of the MK+PGR and PGR100 compositions originates from the non-consumption of C-H of both compositions, that is, there was no conversion to C-S-H for the mechanical behavior be improved in both compositions, since this hydrated phase is the main responsible for the increase in the strength of Portland cement-based composites, as well as for the respective gain in durability.

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